FERMILAB-CONF-04-242-TD

Facility for Chemical Polishing of Superconducting Niobium RF Cavities

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Abstract— **Chemical polishing of surfaces of Nb RF cavities is an important part of cavity fabrication process. Because of the dangerous nature of chemicals involved in the process, safety considerations require thorough design of the facility and of the process. This report describes the major features of the facility prototype built at FNAL including the process description and control system features.**

Index Terms— **Buffered chemical polishing, Process control, Superconducting accelerator cavities, Surface treatment**

I. Introduction

For several years the photoinjector facility at FNAL [1] has been providing valuable information on how the front end of many future large scale accelerating machines, like linear colliders or free electron lasers, can be built. A key issue in theses studies is the manipulation of the electron beam to obtain beam qualities like: total charge, length, shape, and 3-d emittance of an electron bunch. By using a laser driven photocathod-based RF gun, many challenging questions have been resolved. The planned upgrade of the photo-injector test stand [2] will add some new features allowing moving forward and seeking the solution for the remaining open questions. A block scheme of the upgraded facility is show in Fig. 1.

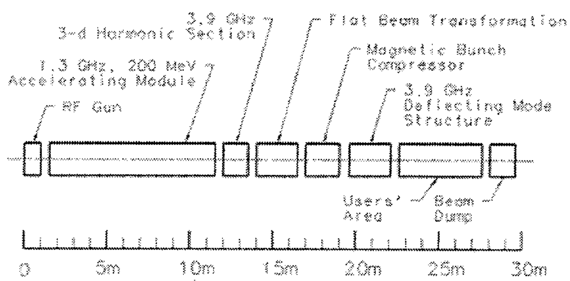

Fig. 1. Photoinjector upgrade layout

Besides the RF-gun, the facility will incorporate a 1.3 GHz superconducting TESLA-type accelerating module [3] bringing the electron energy up to a 200 MeV level, a 3.9 GHz (3-d harmonic) superconducting accelerating section to significantly improve the longitudinal emittance [4], a 3.9 GHz deflecting superconducting section to be used in a "streak camera" mode [5], and beam manipulation and bunch compression magnetic systems.

Out of the three different superconducting RF systems to be used in the facility, at the moment, the two 3.9 GHz systems are under construction at FNAL. As part of cavity fabrication process for both the systems, surface treatment is required to achieve acceptable performance. Although quality of bulk Nb used to build cavities is very good (RRR $\approx$ 400), during the fabrication, the outermost layer of Nb is damaged and contaminated by foreign materials that need to be removed. The chemical treatment (etching) is accepted worldwide as a method to do it [6]. There are two major approaches to chemical etching: Buffered Chemical Polishing (BCP) and Electropolishing (EP). Although recently EP has proven to show better results, FNAL is using the BCP treatment as the primary procedure mainly due to its widely understood process techniques [7] and lower costs.

II. BCP Chemistry and Safety Concerns

BCP uses a mix of three acids: Hydrofluoric HF (49%), Nitric HNO_3 (69.5%), and Orthophosphoric H_3PO_4 (85%). Nitric acid reacts with Nb to form Niobium pent-oxide Nb_2O_5. Hydrofluoric acid reacts with this pent-oxide to form Niobium Fluoride NbF_5, which is hydrosoluble. Orthophosphoric acid serves as a buffer that helps to keep the reaction rate, that depends on the temperature and concentration of Nb in the solution, constant. Although different laboratories use slightly different proportions of the mix, it is quite typical to use a mixture in the volumetric proportions of 1:1:2 (HF : HNO_3 : H_3PO_4). There is a rule of thumb to keep the acid temperature below 20°C and concentration of Nb below 15 g/l. Therefore, a minimum of about one liter of BCP is needed to remove 1 μm of Nb for each 1 m^2 of surface area treated. As a result, for a typical cavity processing cycle, the required amount of polish mix can be more than 100 liters. This translates into a significant attention to issues of personal and environmental safety and results in the concept of a remotely controlled process [7].

The thorough description of the chemical reactions can be found in [8]. One of harmful byproducts of the process is Nitrogen Dioxide NO_2. Five moles of NO_2 evolve for each mole of reacted Nb. With the etching rate of 1 μm/min, about

Manuscript received October 4, 2004.
This work was supported in part by DOE
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25 g (or ~12 liters) of NO_2 are released each minute from 1 m^2 of the cavity surface. Besides NO_2, there is also emission of NO, HNO_3, and HF, which are also considered dangerous pollutants. Although fumes of HNO_3 and HF are released only by evaporation, at 15°C the evaporation rate is 7 g/(h·m^2) for HF and 12 g/(h·m^2) for HNO_3 and concentrations of theses gases in the etching room (60 m^3) can easily exceed the acceptable limit of 2 ppm. In a case of an acid spill, the situation is more severe since the evaporation surface is larger.

The type and amount of gases released during the procedure necessitate the use of proper ventilation and exhaust fume scrubbing to guarantee minimal exposure of personnel and environment to harmful gases.

The acid mix that can't be used for etching any more is usually sent out for utilization by chemical industry. The rinsing water must be neutralized on-site before it can be dumped into a sewage system; for this purpose an appropriate neutralization system must be employed.

Some important features of the facility are introduced to address certain requirements of the process. For example, the cavity, unless working in a class 10 clean room, should not be exposed to air for longer than 1 minute in order to avoid quality degradation, that can be explained by formation of phosphate inclusions, which are hard to remove. Moreover, only well filtered air can enter a treated cavity, and only ultra pure water must be used for rinsing to prevent cavity surface from being contaminated.

III. Facility Layout

Fig. 2 shows a block-diagram of the facility.

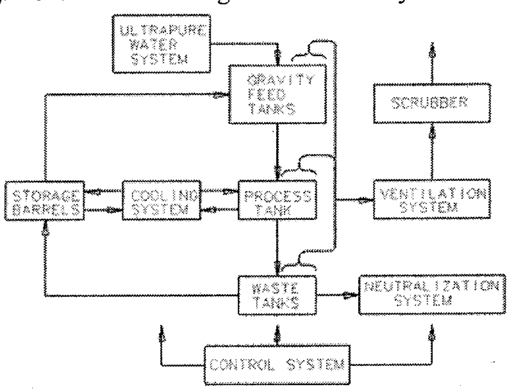

Fig. 2. BCP processing facility: block diagram

The main concept of the design is to have the ability to remotely operate the chemical process from outside the process room where all the acid circuits are confined, thus preventing operators from exposure to acids and fumes. The acid is transported in and out of the room in double contained barrels (HDPE outside, Teflon inside) placed in DOT-approved over-packs (HDPE). The room ventilation is provided by a wet scrubbing system with the efficiency of 95% for NO_2 and 99% for the rest of the dangerous gases. The capacity of the scrubber is sufficient for treatment of one TESLA-type cavity per day and also for manual etching of non-standard parts in acid-filled tanks with an open surface of up to 1 m^2. By using local ventilation hoods and by monitoring the concentration of the pollutants by a gas analyzer, a controlled environment can be created to allow the presence of properly dressed personnel in the room when some small-scale etching is required.

The layout of the process compartment has been developed based on the requirement of process TESLA-type cavities. The size of cavities defines the capacity of all tanks (~250 liters), the height of the room, and the footprint of the associated equipment. Fig. 3 provides an artistic view of the interior of the process room.

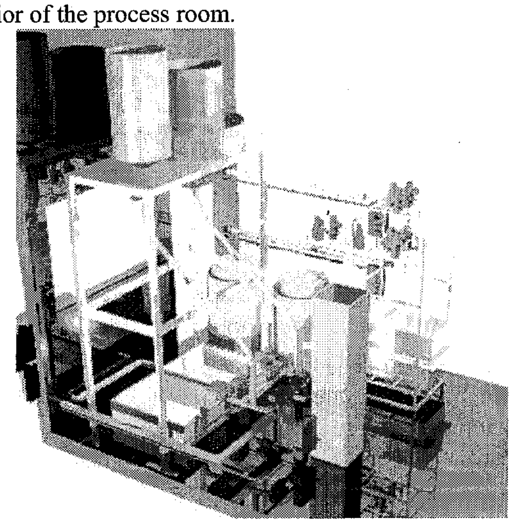

Fig. 3. BCP processing compartment

The room is 4.5 m in length, 3.5 m in width, and 4.5 m in height. It is lined on the inside with PVC sheets to form a watertight container and is equipped with splash-proof doors. Most of the critical components in the room, like barrels and tanks, are provided with secondary containment vessels and the critical piping circuits are equipped with gutters to localize spills if any happen. In a case of a major spill, thanks to a slightly tilted floor, the acid is collected in a sump area with the capacity exceeding 250 liters, located along one of the sides of the processing room.

The hydraulic scheme of the facility is described in [9]. The topology of the hydraulic system was developed to minimize the length of piping, to simplify the handling of the barrels with clean and used acid mix, and to provide enough space for the personnel to safely install and remove cavities from the process tank or to perform maintenance activities like repairs and cleaning. The parts of the system where the acid can't be removed from (e.g. filter and heat exchanger) are separated from the other components by ball valves.

IV. Process Description

There are three major steps of the BCP process: acid cooling, etching, and rinsing. The system preparation and testing as well as post-process clean-up are equally important for the reliable operation of the facility and for stable and reproducible results.

The BCP process is exothermal: for each mole of Nb etched out, 312 kJ of heat are released into the solution. The rate of the chemical reaction increases with the temperature and there can be a process run-out if temperature increases 20°C. Commonly the temperature during the process is kept around 15°C and the acids in the barrel brought into the process room are thoroughly mixed to avoid stratification. The cooling takes place in an immersion style GF CALORPLAST PVDF-made heat exchanger that uses XCELTHERM CF as cooling media. A cooling power of 3 kW at 5°C is provided by a standard chiller filled with propylene glycol. The time needed for cooling is about 2 hours for 250 liters of acid mix.

Gravity feed tanks are used to meet the important criterion of filling the process tank in less than one minute. Since the amount of acid used in the process can be up to 250 liters, using a pump to fill the process tank is not a reasonable alternative. The same idea serves for fast removal of acid or rinsing water out of the process tank. In this case, additional tanks are used that temporary contain acid or rinsing water while it is relatively slowly pumped out of the tanks into an acid storage barrel or neutralization system.

During the process, when the acid reacts with Nb, the heat generated reaches about 500 W for a TESLA-type cavity (treated surface of about 1 m^2). Additional 500 W are lost in the piping used for the acid circulation due to the heat exchange with air. This power can bring the acid temperature close to the threshold of 20°C, and cooling must be used to keep the process under control. This cooling is provided by the cooling system that was use for the initial acid cooling.

Usually the inner and outer surfaces of a cavity need a different treatment. To make it possible, PVDF-made jackets are used to protect inner or outer surfaces when they do not need to be etched. These jackets are filled with ultra pure water circulating through the cooling circuit.

After the etching is completed, the acid in the process tank with the processed cavity inside must be replaced with rinsing water. The rinsing is repeated until it is safe for the personnel to handle the cavity. Fig. 4 shows the expected pH of the rinsing water as a function of the number of rinsing cycles and amount of acid left in the system [10]. At least three rinsing cycles are required before reaching pH > 4, which is considered safe.

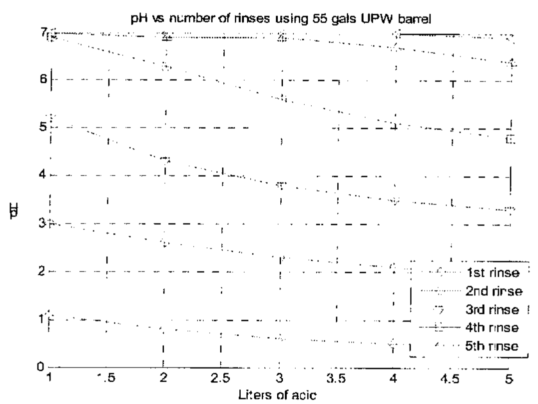

Fig. 4. pH as a function of number of rinses

All the water used for rinsing is pumped into a standard automated neutralization system. A pH meter detects the acidity of the rinsing water and, on the base of these readings, the water is neutralized using a caustic solution or directed to a drain.

Gases and fumes generated during the process are caught by local hoods and removed by a ventilation system for neutralization by a wet scrubber. The ventilation system is configured to prevent the gases and fumes release in the room during the normal operation, to provide optional ventilation capacity for a hood used for manual etching inside the room, and to ensure a quick air exchange inside the room and inside the acid trench in a case of a major spill. The capacity of the ventilation system and the scrubber is 3000 cfm. The configuration of the ventilation system can be changed remotely at different stages of the process by using dumpers.

The major air contaminant concentrations are monitored during and after the process to provide the personnel with information on the air quality. For this purpose a gas analyzer equipped with two gauges sensitive to HF and NO_2 is used.

In order to provide clean air exchange in the room, a pack of EPA filters is installed in the air intake. For additional protection from particle contamination, the gravity feed tanks and the process tank are provided with additional filters to ensure the cleanliness of the air that may enter in the cavity under process.

V. Main Features of the Control System

Due to the aggressive nature of acids and fumes inside the process room, measures must be undertaken to ensure reliable functioning of the involved equipment. We tried to avoid using electrical power circuits inside the room and to protect all electrical and pneumatic circuits from being exposed to the room environment. All the pumps and the valves in the room are pneumatically driven and are controlled by solenoidal valves mounted outside the room. The Polyethylene compressed air lines are traced to the points of use inside PVC piping with junction boxes at the ends. The feedback signal cables are also traced through PVC piping hermetically connected to junction boxes. Between the junction boxes and the sensors or reed switches are enclosed in PE piping so that the wiring is not exposed to the aggressive gases.

A semi-automated operating mode is used to control the process that was broken into several steps. The system performs each step in the automatic mode, but the decision to switch to the next step is generated by an operator. The semi-automated steps of the process are: acid cooling, gravity tank filling, etching, rough rinse, and fine rinse.

The process control is based on a Programmable Logic Controller (PLC) and is monitored by a computer-based Human-Machine Interface (HMI). In this case, any failure of a computer system does not disturb the process. Built-in back-up power capabilities allow safe completion of the process at any stage in a case of a major power failure. Once programmed, the PLC can control the process, watch the system status through a system of gauges, report this status to the operator, and perform other useful functions like statistical analysis etc.

At any time, the facility operator can switch from semi-automatic to manual mode. During the manual mode, the operator can directly control all the components with the use of the HMI by clicking on the icons representing the components on the computer screen. An interlock system prevents accidental or unauthorized access to the manual mode.

A complete description of the control system is found in [11]. Fig. 5 shows control panel with the instrumentation rack.

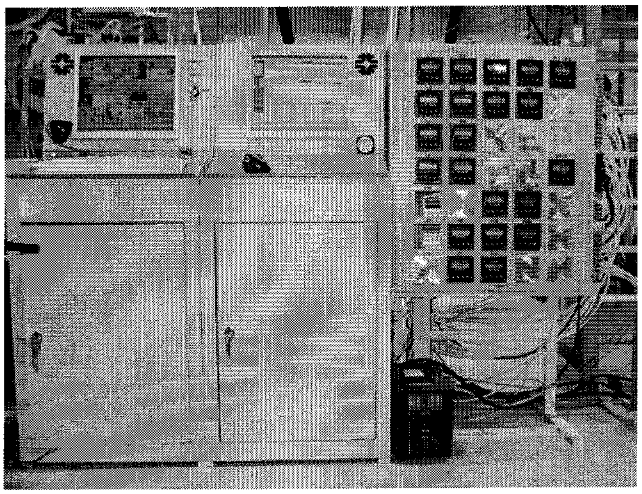

Fig. 5. Control panel

VI. Mockup Test and Future Plans

The BCP facility is to be assembled at ANL, where it is going to be part of the ANL-FNAL SRF Cavity Surface Treatment Facility. Before assembling the facility at ANL, a full-scale mockup has been built and is being tested at FNAL to check the functionality of all sub-systems. During this testing, the main attention points were the available cooling power, the time to fill and discharge the process tank, and the effectiveness of the safety features programmatically embedded in the control system. The tests have shown a process tank filling and discharge time (250 liters) of about 45 seconds that meets the requirements. It takes about 2 hours to cool acid from room temperature to the design level of ~10°C. A temperature rise of about 1°C occurs when the acid is pumped into the gravity feed tank (it takes 10 minutes to pump up 250 liters of acid). This rise is due to heat transfer through the walls of the piping and corresponds to a power loss of about 500 W. The cooling system can remove up to 2 kW of heat generated during the process. The expected power generation is on the level of 1 kW.

The system is equipped with many gauges: GF sensors of temperature, level, flow rate, conductivity, acidity, pressure, help monitoring the process at different stages. The readings of the gauges are used to provide the needed feedback to the control system and to set certain control marks for the interlock system that was designed to protect the personnel and the processed cavity. A complete description of the embedded safety features can be found in [12].

VII. Conclusion

A semi-automated system has been developed to perform buffered chemical polishing of SRF cavities. The system is to be installed at ANL as a part of the ANL-FNAL Superconducting Cavity Surface Treatment Facility.

The full-scale system mockup has been built and is being tested at FNAL. At ANL, the process room has been built to accept the facility and the scrubbing system has been built and tested. After the system is transferred to ANL, extensive personnel training and safety review must happen before first sample etching cycles start.

Meanwhile, the committee on the technology choice for an International Linear Collider has agreed on the superconducting approach for the project. This decision put a solid base to our activities towards SRF technology development, which includes, but is not limited by chemical processing.

Acknowledgment

The present configuration of the system resulted from multistage discussions with those of SRF community who had already experience on buffered chemical polishing, like A. Matheisen of DESY and H. Padamsee of CU, and with those who demanded it, like H. Edwards and L. Bellantoni of Fermilab. Authors also thank A. Rowe (Fermilab) for his help in understanding requirements for the facility, S. Bailin (Panner Sales Co.) for his advices on the equipment choice, and D. Connolly and D. Assell for their persistence and devotion in setting up the mockup.

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